IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Application of

Applicant(s)	: Phelps, et al.
Serial No.	: 10/625,885
Filed	: July 23, 2003
Title	: NON-TOXIC CORROSION-PROTECTION PIGMENTS BASED ON RARE EARTH ELEMENTS
Docket No.	: UVD 0279 IA / UD 267
Examiner	: P. Hailey
Art Unit	: 1755

MAIL STOP AF
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

DECLARATION OF JEFFREY A. STURGILL

Jeffrey A. Sturgill, one of the applicants in the above-identified patent application, declares as follows:

1. I received a B.S degree in Geology from the University of Toledo in 1986. I was employed by the University of Dayton from November 1993 until September 2006. I have been working the area of corrosion-inhibiting pigments since 1996, and in the area of corrosion/materials degradation since 1985.

2. I am familiar with this application as well as the Office Action mailed August 22, 2006, including the rejections made by the Examiner therein. I am also familiar with the references cited by the Examiner in that Office Action including U.S. Patent Nos. 6,200,672 to Tadokoro (treated as equivalent to WO 98/48075), and 5,322,560 to DePue.

3. I prepared solutions using Tadokoro's process and the organic compounds 2-hydroxynicotinic acid, catechol, dextrose, and salicylic acid. Dextrose was used as a

close surrogate to γ-cyclodextrin because γ-cyclodextrin was not readily available. However, I do not believe that this difference would change the results. Salicylic acid was used because it is slightly less soluble in water than 2-hydroxynicotinic acid.

4. The solutions were prepared as follows. A 1:1 molar ratio of ammonium cerium IV nitrate to the organic was mixed together. Specifically, 0.02 moles of the respective organic was dissolved in 50 ml of deionized water. The organics were catechol (2.2 g used), dextrose (3.6 g used), 2-hydroxynicotinic acid (2.8 g used), and salicylic acid (2.8 g used). In a separate beaker, 10.9 grams of ammonium cerium IV nitrate was dissolved in 30 ml of deionized water. The specific organic was then mixed with the 10.9 g of ammonium cerium IV nitrate dissolved in 30 ml of water.

5. When the catechol solution was slowly added to the ammonium cerium IV nitrate solution, some bubbling occurred at the surface. As the 1:1 molar mixture was reached, the solution gradually became dark and cloudy. Conversely, when the ammonium cerium IV nitrate solution was slowly added to the catechol solution, no bubbling was observed, but the solution also became dark and cloudy as the 1:1 molar ratio was reached. The catechol solution was a tan brown to begin with. The starting color of the ammonium cerium IV nitrate solution was orange. The ending color of the mixed solution was orange-brown once the 1:1 ratio was reached. The solution was an opaque black after mixing for 24 hours.

After the solutions had been mixed for 24 hours, the opaque black solution was filtered. The supernatant was always black (from the extremely small particles formed during the reaction passing through the Catalog No. 28320-085 VWR Brand qualitative filter paper), but enough solids were collected for a compositional analysis. The black solid was analyzed for tetravalent cerium using the procedure described on page 246 of Reagent Chemicals - Specifications and Procedures 10th edition by the ACS Committee on Analytical Reagents, 2006. The analysis was performed by the Quality Control group of GFS Chemicals, which has many years in certifying the characteristics of tetravalent cerium chemicals. Specifically, 2.0 grams of the black solid was weighed to the nearest 0.1 mg. The sample was dissolved in 2 ml of sulfuric acid and titrated with a redox

probe. No cerium in either the tetravalent or trivalent oxidation state was found in the sample. Given the black color of the sample (tetravalent and trivalent cerium compounds are typically white, yellow, or orange in color), and the results of this ACS-approved analysis, it is believed that the solid formed via the Tadokoro et al. procedure is elemental carbon.

6. When the dextrose solution was slowly added to the ammonium cerium IV nitrate solution, some bubbling occurred at the surface. As the 1:1 molar mixture was reached, the solution became dark orange. Conversely, when the ammonium cerium IV nitrate solution was slowly added to the dextrose solution, no bubbling was observed, but the solution also became dark orange as the 1:1 molar ratio was reached. No matter what order the reagents were mixed, the solution, which was dark orange initially, became clear after 5 minutes of mixing. The solution was still clear after 24 hours of mixing, and no precipitate was observed.

The dextrose-containing solution was evaporated down over low heat overnight. In the morning there was only 20 ml of water left in the beaker (from the original 80 ml), and solid material had not appeared. Even assuming the end result was a cerium IV-dextrose complex, the aqueous solubility is so high that it would fall well outside the claimed solubility range. It would not be usable as a pigment material.

7. When the 2-hydroxynicotinic acid was added to the ammonium cerium IV nitrate, not all of the 2-hydroxynicotinic acid dissolved. It was necessary to heat the solution to approximately 60°C in order to get all of the 2-hydroxynicotinic acid into solution. Upon mixing, the solution turned black. A brownish precipitate appeared after 24 hours when the acid was completely dissolved in water (i.e., the bath at approximately 60°C). If the bath was at room temperature (i.e., the acid was not totally dissolved in water), the brownish precipitate started appearing within 5 to 10 minutes, and after 24 hours, the reaction was complete. An analysis was performed on the precipitate formed from the dissolved acid. A copy of the test results for cerium IV is attached hereto as Exhibit A. The summary of the results states TITER - missing EP (endpoint). Accordingly, there is no cerium IV in the sample. The titrimetric curve is also shown. If cerium IV was

present, there would have been a noticeable peak. As can be seen, no peak is present. The sample was also tested for cerium III, and the results indicated that cerium III was present.

The sample which was not analyzed (where the acid was not totally dissolved) will not have cerium IV in it, because the reaction was much more vigorous - i.e., there was much more bubbling. In other words, the undissolved organic was even more of a reducing species than the dissolved organic.

8. When the salicylic acid was added to the ammonium cerium IV nitrate, not all of the salicylic acid dissolved. It was necessary to heat the solution to approximately 60°C in order to get all of the salicylic acid into solution. A brownish precipitate appeared after 24 hours when the acid was completely dissolved in water (i.e., the bath at approximately 60°C). If the bath was at room temperature (i.e., the acid was not totally dissolved in water), the brownish precipitate started appearing within 5 to 10 minutes, and after 24 hours, the reaction was complete. An analysis was performed on the precipitate formed from the dissolved acid. No cerium IV was present. The sample was also tested for cerium III, and the results indicated that cerium III was present.

The sample which was not analyzed (where the acid was not totally dissolved) will not have cerium IV in it, because the reaction was much more vigorous - i.e., there was much more bubbling. In other words, the undissolved organic was even more of a reducing species than the dissolved organic.

9. The reaction involving catechol resulted in a solid. The solid produced was analyzed for cerium content using ACS-specified procedures, and neither tetravalent or trivalent cerium was determined to be in it. This solid is likely elemental carbon. Two other compounds (2-hydroxynicotinic acid and salicylic acid) produced precipitates that contained cerium III. The other compound (dextrose) yielded a highly water-soluble complex that would be unsuitable for use as a pigment due to its affinity for water. None of the reactions produced a cerium IV complex.

The process described in Tadokoro is non-enabling for making a tetravalent cerium complex, and it would take undue experimentation to produce a tetravalent cerium complex using Tadokoro's process.

The declarant further states that the above statements were made with the knowledge that willful false statements and the like are punishable by fine and/or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of this application or any patent resulting therefrom.

Date: Nov. 22, 2006

Jeffrey A. Sturgill

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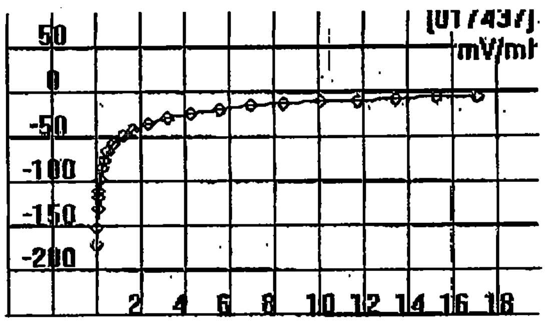

EXHIBIT
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